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Statistical analysis of the effect of operating parameters on acid mist generation in copper electrowinning

Reza Al Shakarji [a], Yinghe He [a,*], Simon Gregory [b]

[a] School of Engineering and Physical Science, James Cook University, QLD 4811, Townsville, Australia
[b] Xstrata Technology, Copper Refinery Limited, Hunter St, QLD 4811, Townsville, Australia

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ABSTRACT

Acid mist is generated during the final stage of hydrometallurgical metal refining processes including the electrowinning of copper. In this study, the effect of five process parameters and their interactions on the amount of acid mist generated is analysed quantitatively. The amount of acid mist generated was measured under 32 different operating conditions. It was found that solution's temperature and mist suppressant chemical FC-1100 had significant effect on the amount of acid mist generated. More than 90% of the variations in the acid mist generation can be explained by changes in these two parameters and their interaction. To a lesser extent, electrical current density and solution acidity also affected the total amount of acid mist generated. The anode's age and most of the 3, 4, and 5-way parameter interactions were found to have negligible influence on the amount of acid mist. Overall, acid mist was found to increase with temperature and current density. In contrast, increasing the viscosity of the solutions tends to decrease the amount of acid mist.

1. Introduction

Electrowinning is an electrochemical process that is used to extract metal from its solution, and is extensively used in the production of copper. More than 20% of the world's primary copper is produced through electrowinning (Davenport et al., 2002).

In the electrowinning of copper, a direct electrical current is passed between an anode and a cathode that are submersed in a copper-rich solution (Robinson et al., 1994). At the inert anode, water molecules are electrolysed and oxygen bubbles are formed on the surface of the anode. These oxygen bubbles grow and eventually detach from the surface and rise in the bulk of the solution. These bubbles burst at the free surface of the solution and produce highly acidic droplets; of which the fine ones become airborne and form an acid mist throughout the tankhouse of the electrowinning plant.

Acid mist is highly corrosive and results in the corrosion of cathode plates, anode's hanger bar, tankhouse equipment and building structures. Acid mist also poses a serious health hazard and causes extreme discomfort to the skin, eyes and respiratory systems of the tankhouse workers (HSIS, 2009). The Occupational Safety and Health Administration (OSHA) recommends a time-weighted average (TWA) exposure limit of 1 mg of sulphuric acid per m^3 of air, and a short term exposure limit (STEL) of 3 mg m^{-3} (OSHA, 2003).

There have been many attempts to eliminate or minimize acid mist in copper electrowinning operations (Mella et al., 2006). Polyethylene balls, suction hoods, mats, brushes and wipers, chemical reagents and forced ventilation are examples of such attempts (3M, 2007; Davis and Eng, 2002; Hooper, 2008; Mella et al., 2006; Sunwest, 2004). Qualitatively, the use of chemical reagents such as FC-1100, Mistop, Dowfroth, and alkylated ethoxylates has been rated as the most effective method of suppressing acid mist (Bender, 2010). However, there have been no systematic studies to quantitatively compare the effect of different operating parameters, including the use of a chemical reagent, on the amount of acid mist generated. Most of the published works to date, have only examined the effect of one or two parameters individually without considering any possible interaction effects on the amount of acid mist (Alfantazi and Dreisinger, 2003; Cheng et al., 2004; Hosny, 1993; San Martin et al., 2005a,b; Sigley et al., 2003).

This paper examines, quantitatively, the relationship between the amount of acid mist generated and five operating parameters. These parameters are the age of the anode, electrical current density, solution temperature, sulphuric acid concentration of the solution, and the presence of a typical chemical mist suppressant (i.e. FC-1100). The results are useful for the design of more efficient methods or systems for acid mist minimization at electrowinning tankhouses.

* Corresponding author. Tel.: +61 7 47814270; fax: +61 7 47816788.
E-mail address: yinghe.he@jcu.edu.au (Y. He).

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2. Methodology

2.1. Equipment set up

The copper electrowinning process was replicated in a bench-scale cell. This cell (C2 in Fig. 1) was constructed of 10 mm thick clear acrylic and had a capacity of 6 L. During each test, electrochemical reactions resulted in continuous copper depletion and acid generation in the solution. Therefore, a peristaltic pump was utilized for gradual and continuous addition of fresh solution, from C1 container to C2, to maintain consistency in the composition of the solution. A horizontal slit in the side of C2 was utilized to keep the level of the solution in the container constant.

Four submersed heaters were placed in the corners of C2 to keep the solution at a constant temperature for the duration of each test. To replicate industrial operations, Pb–Ca–Sn alloy and 316 L stainless steel were used as anode and cathode, respectively (Houlachi et al., 2007). For each set of experiments a fresh batch of solution was synthesized that contained 45 g L^{-1} Cu, 15 mg L^{-1} Guar gum, 20 ppm Cl and 100 ppm Co. The synthesized solution was similar to the electrolyte solutions found in most copper electrowinning tankhouses worldwide (Robinson et al., 1994). The sulphuric acid concentration in the solution, however, was one of the five selected variables and its concentration differed from that of a typical copper electrolyte solution.

Nitro cellulose filters were used to capture acid mist. These filters were 47 mm in diameter and had a pore size of 0.45 μm which ensured the capture of very fine acidic droplets. For each experiment, a fresh filter paper was installed at 45 mm above the free surface of the electrolyte solution inside the electrowinning cell (C2). The filter was held by an inverted funnel (F in Fig. 1) and connected to a vacuum pump (VP) via a pneumatic tube. For precise air flow measurements, the drawn air was dehumidified by passing it through an enclosed flask that contained silica beans (D). A flow meter (FM) with a built in valve was installed on the pneumatic tube to ensure a constant air flow of 5 L min^{-1} through the filter for the duration of each experiment.

At the end of each test, the used nitro cellulose filter was removed from the cell and placed in 25 mL of deionized water and stirred for about 60 min. The pH of the solution was then measured and the amount of the captured acid was calculated as grams of sulphuric acid per cubic meter of air drawn through the filter.

2.2. Experimental design

The main goal of the present work was to compare, quantitatively, the effect of different operating parameters on the amount of acid mist generated during a typical copper electrowinning process. To fully explore the main effect of each individual parameter as well as any possible interaction effects, the 2 K factorial method was utilized to determine the required experimental conditions (Montgomery, 2005). In this method, the examined parameters are tested at two levels (low and high). The bigger the difference between the low and high levels of a parameter, the more reliable its effect measurements would be (Montgomery, 2005). Table 1 illustrates the examined parameters and their low and high level values.

The values shown in Table 1 were selected so that the midpoint between low and high levels of each parameter represented the typical value used in most copper electrowinning tankhouses (Houlachi et al., 2007). For example, the temperature of the electrolyte solution is usually kept at about 45 °C. Thus, the low and high limits of temperature for the experiments were set at 30 °C and 60 °C, respectively.

Based on the 2 K factorial method, 32 parameter combinations were required to fully examine the effect of five parameters at two levels. For reliable data analysis, a minimum of two replicates were needed for each test condition. Hence, 64 tests were conducted to determine the influence of each parameter on the generation of acid mist and also to identify any significant interactions amongst the selected parameters.

3. Results and discussion

3.1. Descriptive statistical analysis

In all the 64 experiments at 32 different operating conditions, the least amounts of detected acid mist were 0.03 and 0.02 mg acid m^{-3} air. These two tests were repeats, conducted with an old anode at low current density in a solution with low acidity, low temperature and high concentration of FC-1100.

The highest amounts of detected acid mist were 114.4 and 117.1 mg acid m^{-3} air. These two repeats were conducted with a new anode at high current density in a solution with low acidity, high temperature and no FC-1100.

The test results proved to be highly repeatable. The average relative error between the repeats of medium–high acid mist concentration tests was 18%. The smallest relative error was 0.001%. For the very low acid mist concentration tests, however, the average relative error between the repeats was 41%. This high relative error was essentially due to the resolution limit of the measurement method.

To distinguish the effect of individual parameters, the average amount of collected acid mist for each level of a parameter was calculated. The bar graph in Fig. 2 illustrates the results where each bar represents the average amount of acid mist collected from 32 independent experiments.

In Fig. 2, it can be seen that highest amounts of acid mist were generated when the solution was kept at high temperature or when no FC-1100 was added to the electrolyte solution. To a lesser extent,

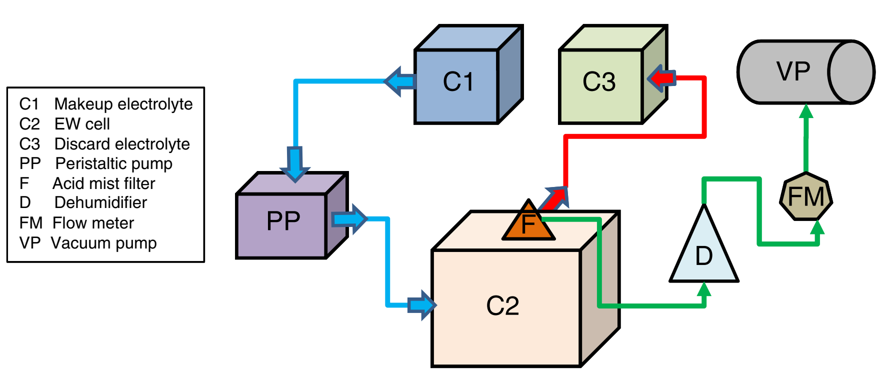

Fig. 1. A schematic view of equipment setup.

Table 1
The selected test variables and their values.

No	Examined parameter	Low	High
1	Anode age (months)	0	6
2	Current density (A m^{-2})	200	400
3	Temperature (°C)	30	60
4	Acidity (g L^{-1})	100	250
5	FC-1100 (ppm)	0	30

high current density, low sulphuric acid concentration in the solution and new anode also favoured higher acid mist generations.

High temperature solutions produced the most amounts of acid mist. Temperature is known to affect the rheological properties of fluids. Surface tension and viscosity of the electrolyte solution were known to strongly influence the final size and the burst process of the oxygen bubbles (Xie et al., 2009; Xu et al., 2009). Thus, to find an explanation for the strong influence of temperature on acid mist, the effects of temperature on both the surface tension and the viscosity of the solution were evaluated and the results are discussed in detail in Section 3.3.

The second highest amount of acid mist belonged to the tests where no FC-1100 was added to the solution. The addition of 30 ppm of FC-1100 reduced the acid mist amount from 47.2 to 7.3 mg acid m^{-3} air. As will be seen later, this reduction in the amount of acid mist in the presence of FC-1100 is believed to be caused by a change in the burst mechanism of bubbles at the free surface of the solution through changes in the surface viscosity and surface elasticity.

The third highest acid mist concentration belonged to high current density experiments. Based on Faraday's law, electrical current is directly related to the rate of chemical reactions that occur during an electrochemical process (Harris, 2007). Decomposition of water molecules at the surface of the anode is the main anodic reaction that takes place during the copper electrowinning process. Therefore, based on Faraday's law, doubling the electrical current density will double the total volume of the generated oxygen bubbles.

Our previous experimental work suggested that changing the electrical current density had negligible effect on the final sizes of the bubbles that detached from the anode (Al Shakarji et al., 2010). Consequently, doubling the current density will approximately double the number of oxygen bubbles generated. This increase in the number of bubbles (per unit time) with current density results in a net increase in the number of bubbles that burst at the free surface of the solution which in turn produces a higher number of airborne acidic droplets (i.e. acid mist) per unit time per unit surface area of the solution.

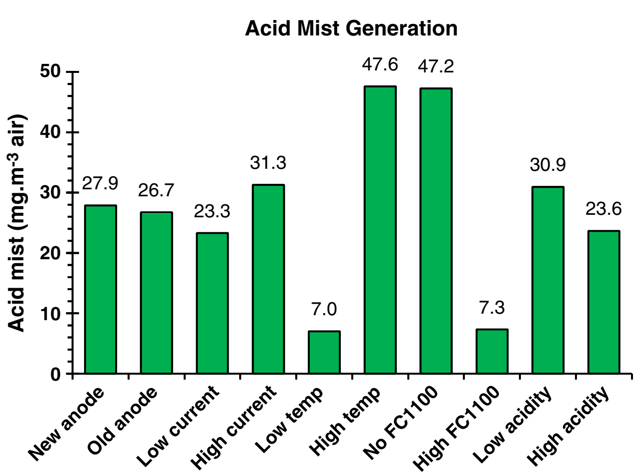

Fig. 2. Mean acid mist generation at different operating conditions.

3.2. Quantitative statistical analysis of test parameters on acid mist generation

A 5-way ANOVA analysis was conducted on the raw experimental data to determine the full effect of test parameters on the amount of acid mist generated. This analysis returned p values less than 0.05 (i.e. significant at 95% level) for 18 of the 32 possible parameter combinations. Cohen classifies the magnitude of a parameter's effect into three categories of small, medium and large (Cohen, 1988). These categories correspond to R^2 values of 0.01, 0.09 and 0.25, hence accounting for 1%, 9% and 25% of the total variance, respectively (Cohen, 1988). To quantify the influence of the aforementioned 18 conditions on acid mist generation, the R^2 value was calculated for each case based on its Pearson's r value (Field, 2005). The top ten cases (with respect to R^2) are shown in descending order in Fig. 3.

The quantitative analysis (Fig. 3) shows that temperature, FC-1100 and the interaction of these two parameters are the most influential parameters in determining the amount of acid mist generated. To a lesser degree, current density and solution acidity also affected the generation of acid mist. Anode's age with an R^2 value of 8×10^{-5} was determined to be a parameter with negligible effect on the amount of acid mist generated.

The ANOVA analysis returned an overall adjusted R^2 value of 0.986, which meant 98.6% of the variation in acid mist recorded in the series of experiments could be explained by changes in the five parameters. The uncontrolled variables and instrumentation errors accounted for only 1.4% of the measured acid mist variations which implied the experiments were conducted at a highly controlled environment.

The influence of individual test parameters as well as that of important interactions (shown in Fig. 3) on acid mist is discussed in more details in the following sections.

3.2.1. Effect of temperature and FC-1100 on acid mist

Based on Cohen's classification, both temperature (T) and FC-1100 (S) parameters with R^2 values of 0.378 and 0.366, respectively, proved to have large effects on the amount of acid mist generated. The interaction of these two influential parameters (T*S) with an R^2 value of 0.159 had a medium effect on acid mist. These effects can be seen graphically in Fig. 4.

Fig. 4 also shows that the presence of FC-1100 in the electrolyte solution strongly influenced the effect of temperature on the amount of acid mist generated. In the absence of FC-1100, a 30 °C increase in the solution temperature increased the amount of acid mist by almost 67 mg m^{-3} of air whereas in the presence of FC-1100 the same increase in the solution temperature resulted in only 14.3 mg more acid mist per m^3 of air.

The vast difference in the slopes of the two lines in Fig. 4 indicated the strong interaction between temperature and FC-1100. The T*S interaction alone accounted for nearly 16% of the variations seen in the amount of acid mist generated. Overall, temperature (T), FC-1100 (S), and T*S have a combined R^2 value of 0.903 which means that

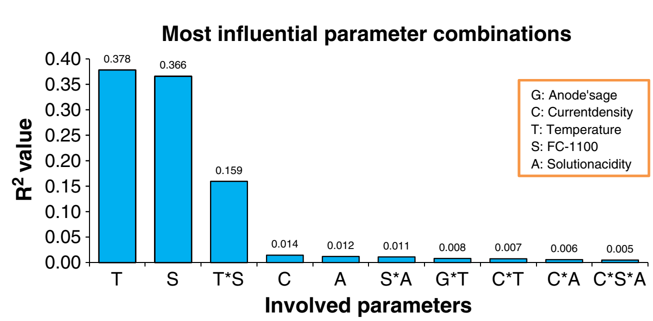

Fig. 3. Quantified influence of different parameters and parameter combinations on the amount of acid mist generated.

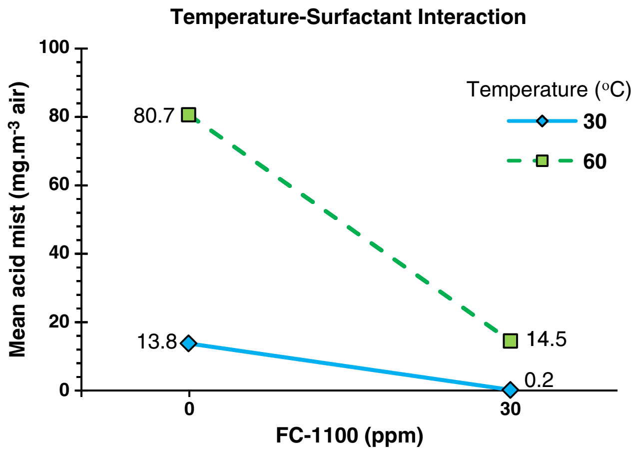

Fig. 4. The temperature–FC-1100 interaction based on mean acid mist values.

changes made in these three parameters are sufficient to explain more than 90% of the variations seen in the amount of acid mist generated.

3.2.2. Effect of current density on acid mist

Current density with an R^2 value of 0.014 was the fourth influential parameter in the amount of acid mist generated. In absolute terms, the effect of current density on acid mist is much less than that of temperature and FC-1100 and based on Cohen's classification the effect of current density is considered small. Nevertheless, in relative terms, current density is an important factor as the amount of acid mist generated at 400 A m^{-2} was 34% more than that generated at 200 A m^{-2} (Fig. 2).

To compare the magnitude that current density and FC-1100 affect the amount of acid mist generated, the averaged acid mist measurements were plotted at different current densities with or without the presence of FC-1100. The results are shown in Fig. 5.

Fig. 5 confirms again a substantial reduction in acid mist at both low and high current densities when FC-1100 is added to the electrolyte solution. Since there is no significant difference in the slope of the two lines in Fig. 5, it means that current density has no significant influence on the effect of FC-1100 on acid mist generation, i.e. little interaction effect between the current density and FC-1100.

Fig. 5 also shows that, regardless of the presence of FC-1100, an increase in current density increases the amount of acid mist generated. However, the acid amount did not double when the current density was doubled. This is because acid mist is produced from the burst of bubbles that ejects droplets into the air of which some become airborne. The amount of mist generated from the simultaneous bursts of two neighbouring bubbles is known to be less than the sum of mist amount from the bursts of two individual bubbles separately, due to interferences between the bursts of the bubbles. At a higher current density, the likelihood for a number of neighbouring bubbles to burst simultaneously is higher. Consequently, even though the number of oxygen bubbles must be doubled when the current density is doubled, based on Faraday's Law and that the bubble size does not change with current density, the acid mist amount is expected to be less than double.

3.2.3. Effect of solution acidity on acid mist

The ANOVA analysis returned an R^2 value of 0.012 for the solution acidity which meant this parameter was the fifth most influential parameter in acid mist generation, which is comparable to that of the current density (0.012 vs 0.014). This means that the acidity of the solution is approximately as influential as the current density in acid mist generation.

The C*A interaction is listed as one of the top ten influential parameters (Fig. 3). This interaction is confirmed from the considerable difference in the slope of the two lines shown in Fig. 6. For high acidity solutions, doubling the current density from 200 to 400 A m^{-2} only resulted in 13% increase (equivalent to 0.087 standard deviations) in the amount of acid mist. In contrast, doubling the current density in low acidity solutions resulted in a staggering 54% increase (equivalent to 0.395 standard deviations) in the amount of acid mist.

Fig. 7 shows the effects of solution temperature and acidity on the acid mist amount. It again confirms the profound effect that temperature has. Further, the two lines in Fig. 7 have similar slopes (1.46 for low acidity solutions and 1.24 for high acidity solutions), which suggests that the interaction between temperature and acidity is negligible.

The plots in both Figs. 6 and 7 reveal an interesting phenomenon that low acidity solutions consistently resulted in higher amount of acid mist than high acidity solutions did, regardless of the applied current density and temperature. This apparent counter-intuitive result will be explained in detail in Section 3.3 below.

3.3. The role of surface tension and viscosity in acid mist generation

The previous two sections presented statistical observations on the influence of operational variables and their interactions on the amount of acid mist generated. This section offers mechanistic explanations to the results obtained in this investigation.

3.3.1. Effect of surface tension

Qualitatively, temperature and FC-1100 are both known to affect the surface tension of a solution. To quantify their effect on the

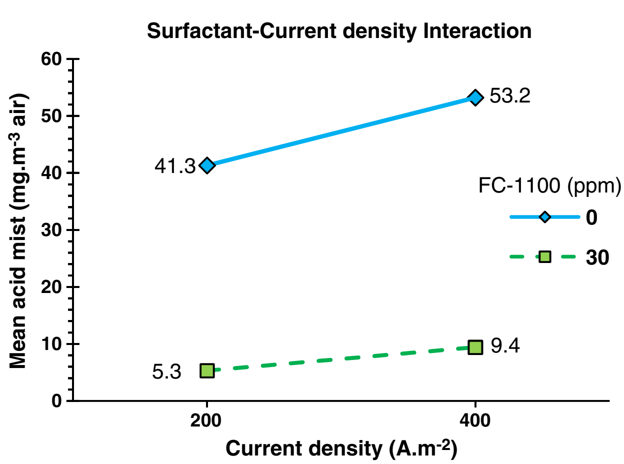

Fig. 5. The effect FC-1100 on acid mist at different current densities.

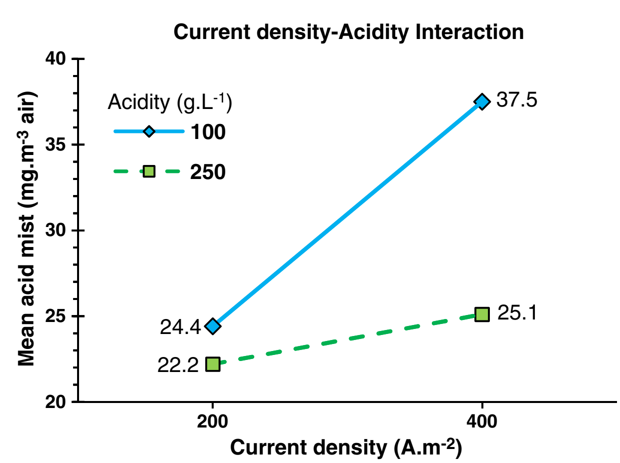

Fig. 6. The effect of solution acidity on acid mist at different current densities.

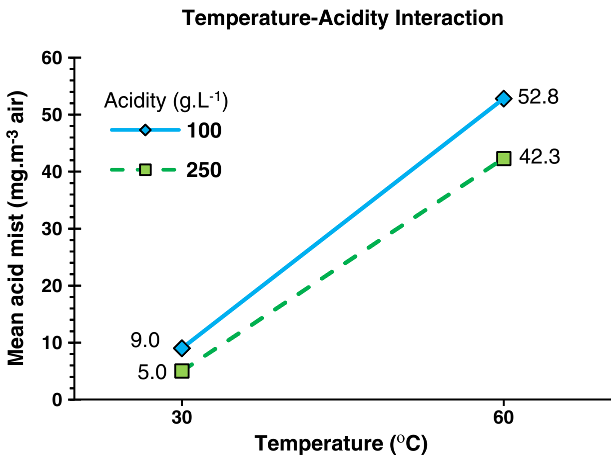

Fig. 7. The effect of solution acidity on acid mist at different temperatures.

electrolyte, the surface tension of 7 samples was measured by the Wilhelmy's plate method at two different temperatures. The averaged results are shown in Fig. 8.

It can be seen that temperature strongly influences the surface tension of the electrolyte in the absence of FC-1100. On average, the surface tension decreases by 32% when the temperature is raised from 30 to 60 °C in the absence of FC-1100. In contrast, in the presence of FC-1100 at 30 ppm, the temperature change has a negligible effect (less than 3%), as shown in Fig. 8. These observations are similar to those seen in Fig. 4 in that, in the absence of FC-1100, the difference caused in the amount of acid mist by temperature change is much larger. Both figures demonstrate a strong interaction between temperature and FC-1100.

It is important to note that, while both increasing solution temperature and addition of FC-1100 cause a significant decrease in the surface tension of the electrolyte, their effect on acid mist generation is the opposite. For example, at 30 °C the addition of 30 ppm FC-1100 reduces surface tension to 44 mN m^{-1} and results in 0.41 standard deviations reduction in acid mist generation. Increasing the temperature from 30 to 60 °C, in the absence of FC-1100, also reduces the surface tension to almost the same value (43 mN m^{-1}). However, this has resulted in an increase of 2.01 standard deviations in acid mist amount.

In the absence of a surface active agent, lower surface tension or lower surface energy, means lower amount of energy required for the generation of new surfaces. In other words, at a lower surface tension, it is more likely to generate higher number and smaller size of liquid droplets from the burst of bubbles. In addition, previous studies also show that the amount of liquid droplets produced from the burst of gas bubbles increases exponentially with decreasing diameter of the bubbles (Liow et al., 2007; Liow and Gray, 1996). That is, the burst of smaller gas bubbles produces more liquid droplets, or mist. In this study, when the temperature is increased, the surface tension is reduced. This has resulted in not only an increase in the likelihood to produce a higher number and smaller size of liquid droplets, but also a reduction in the size of the oxygen bubbles detaching from the anode, causing an increase in the amount of acid mist produced.

In contrast, the reduction in surface tension from the presence of surface active agents not only reduces the final bubble sizes but also changes the bubble burst mechanism at the free surface of the solution. When a bubble rises through the bulk solution and reaches the free surface of the solution, a thin film is produced in the top of the bubble. The stability and lifetime of this thin film is influenced by a number of factors such as surfactant concentration, surface diffusion, surface tension gradient, and drainage rate (Pugh, 1996). Generally, drainage rate decreases with the increase in bulk viscosity, surface viscosity and surface elasticity of the solution (Pugh, 1996). The latter two factors can be increased significantly by the presence of surfactant molecules at the liquid–gas interface (Pugh, 1996). Therefore, it is proposed that the presence of FC-1100 molecules in the solution reduces acid mist mainly via its effects on the thin film drainage rate through a change in the surface viscosity and surface elasticity rather than its effect on final bubble sizes. The slower film drainage and higher surface elasticity in the solutions containing FC-1100 result in the generation of a smaller number of airborne acid droplets (i.e. lesser amounts of acid mist).

3.3.2. Effect of viscosity

To investigate the counter-intuitive inverse relationship between the solution acidity and the amount of acid mist generated shown in Figs. 6 and 7, the viscosity of 12 electrolyte samples (7 low acidity and 5 high acidity solutions) was measured at three different temperatures. The averaged viscosity measurements for the two types of the electrolyte solution are shown in Fig. 9.

Fig. 9 shows as expected that the viscosity of a solution increases with the increase of its acid concentration but decreases with an increase in temperature. The results shown in Figs. 6 and 7, that is, more acid mist is generated by low acid concentration solutions regardless of the applied current density and temperature, can be

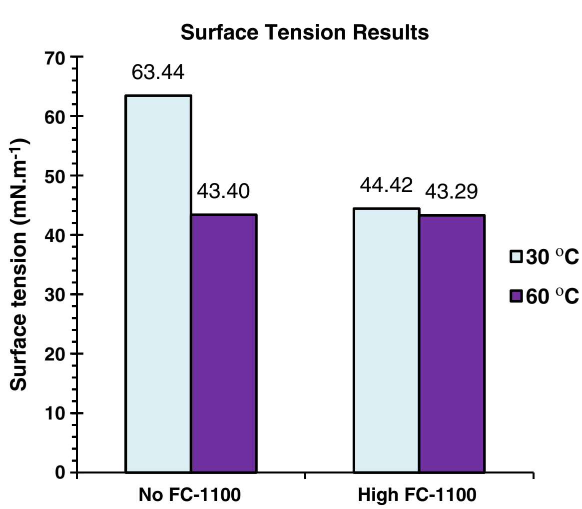

Fig. 8. The effect of temperature and FC-1100 on surface tension.

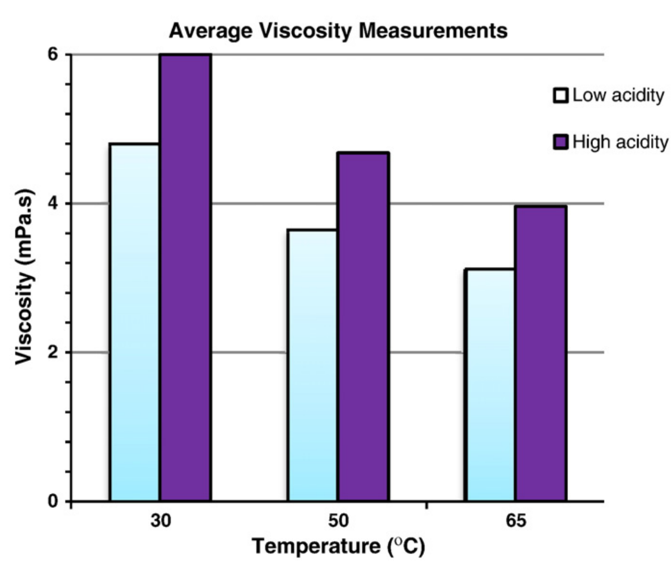

Fig. 9. The effect of solution acidity on its viscosity at different temperatures.

explained by that the amount of acid mist generated is inversely related to the viscosity of the solution.

While no quantitative relationships between the liquid viscosity and the droplet size and size distribution from the burst of bubbles can be offered at present, it is certainly true qualitatively that, the burst of more viscous liquid films or bubbles will produce less number, but in larger sizes, of droplets. This is simply because more viscous liquid films are more difficult to break-up. Further, based on Stokes' law, the terminal velocity of a rising bubble in a liquid is inversely related to the viscosity of the liquid due to the higher drag force exerted on the bubble by the surrounding liquid. As such, the average ascending oxygen bubble velocity in a more viscous liquid is lower than that in a less viscous liquid. Bubbles reaching the free surface at a lower speed would have less dynamic energy to "throw" the droplets from the bubble burst and make them airborne. The results shown in Figs. 6 and 7 imply that, at the high acidity level (high viscosity), the number of electrolyte droplets that become airborne is much less than that from the low acidity level.

It must be noted, however, that the substantial increase in acid mist with increasing temperature is not solely due to the decrease in viscosity. Based on Figs. 7 and 9 it can be seen that a 20% decrease in viscosity due to lower acid concentration in the solution results in 0.22 standard deviations increase in acid mist whereas the 34% decrease in viscosity due to temperature rise results in 1.22 standard deviations increase in acid mist. This significant increase must be attributed to the compound effect of a simultaneous decrease in both the viscosity and surface tension of the solution caused by the temperature increase.

4. Conclusions

The effect of five different operating parameters on acid mist generation was analysed using a full matrix experimental design. The temperature of the solution and the presence of FC-1100 in the solution proved to be the most influential parameters in the amount of acid mist generated. More than 90% of the variations in the acid mist generation can be explained by changes in the two parameters and their interaction.

To a lesser extent, electrical current density and solution acidity also affected the total amount of acid mist generated. Anode's age and most of the 3, 4, and 5-way parameter interactions were found to have negligible influence on the amount of acid mist.

Overall, acid mist was found to increase with temperature and current density. In contrast, addition of FC-1100 to the solutions decreased the amount of acid mist. However, it is critical to note that it is the ability of FC-1100 to increase the surface elasticity and surface viscosity, not its ability to reduce surface tension, that is responsible for the reduction of acid mist generation. The bubble burst mechanism at the free surface of the solution, which is mainly influenced by surface elasticity and surface and bulk viscosity of the solution, proved to be a critical factor in the amount of acid mist generated.

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